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Production of a Sustainable Paving Material through Chemical Recycling of Waste PET into Crumb Rubber Modified Asphalt

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Abstract

Plastic materials such as polyethylene terephthalate (PET) are extensively used in manufacturing drinking bottles, food packaging and many other forms of plastic products. However, the inappropriate disposal of large amount of PET waste may cause severe environmental problems. Using PET derived products as a performance-enhancing additive for asphalt can be one of the alternatives to recycle and reuse this waste material. Similarly, disposal of waste vehicle tyres is also a challenging environmental problem. Research has shown that the use of crumb rubber (CR) from waste tyres in asphalt helps improve some of its rheological properties but is often plagued with other concerns such as low storage stability. The main objective of this study is to investigate the feasibility of using the waste PET additives, derived through an aminolysis process, to improve the storage stability and rheological performance of crumb rubber modified asphalt (CRMA). Both the rheological and chemical properties of the asphalt binders collectively modified with PET additives and CR were investigated. It was found that the incorporation of PET based additives to CRMA improved the storage stability of the asphalt binders by at least 30% at low additive dosage and increased the viscosity by around 20%. In addition, the rutting and fatigue resistances of the modified binders were seen to be improved. Overall, the results indicated that the collective usage of waste PET derived additives and scrap tyre rubber in asphalt not only helps recycle waste plastic and rubber, but also improves the engineering properties of asphalt pavement.

Keywords: Waste PET; Crumb Rubber; Recycling; Modified Asphalt; Sustainable Paving

1. Introduction

The widespread production and consumption of plastics over the past decades have led to huge amount of undisposed or inappropriately disposed waste plastic materials. Along with this, the increasing cost, decreasing space for landfills and corresponding environmental pollution concerns have forced policy makers and researchers to look for alternative solutions for waste plastic disposal (Zia et al., 2007). Thermoplastics, the most common type of plastics, constitute 80% of total polymers produced worldwide and are used for many different applications such as beverage packaging, textile fibres, construction and coatings (Dewil et al., 2006). The thermoplastic polyester industry, largely represented by polyethylene terephthalate (PET), makes up about 18% of world's plastic production and is ranked the third after polyethylene (PE) and polypropylene (PP). According to the report by the National Association of PET Container Resources, approximately 5,971 million pounds of PET bottles were sold into the marketplace in the US in 2015. However, only approximately 30.1% of the total PET sold (i.e., 1,797 million pounds) were collected for recycling (NAPCOR, 2015).

Similar to the condition of waste plastic, the volume of waste tyres has increased globally in recent years with the rapid industrialization and development of the automobile industry. Thus, the recycling and reuse of crumb rubber (CR) produced from automotive and truck scrap tyres have become significantly important to the environment (Presti et al., 2013). One of the best applications of CR is to use it as an asphalt modifier. A significant amount of studies have been carried out to assess the performance of CR modified asphalt (CRMA). It has been generally reported that using CRMA can provide various benefits to pavements, such as better fatigue cracking and rutting resistance, improved durability and lower maintenance costs (Guo et al., 2017; Sienkiewicz et al., 2017; Yu et al., 2017, Yu et al., 2014). However, the storage stability of asphalt-rubber blends at high temperature remains a concern for asphalt producers, especially when high percentage of CR is used (Navarro et al., 2004; Kim and Lee, 2013).

Several studies have been conducted to use chemical additives to improve the storage stability of CRMA. In one study, reactive polyoctenamer and cross-linking agents were applied to improve the anchoring of CR in asphalt and improve the storage stability of the CRMA (Padhan et al., 2017). In another study, SBS and sulphur were used as modifiers to improve the storage stability and tenacity of CR modified asphalt (Zhang et al., 2015).

The main objective of this study is to investigate the feasibility of applying the additives, derived from chemically treated waste PET, to improve the storage stability and rheological performance of CRMA. To achieve this objective, a chemical recycling method based on aminolysis was first applied to degrade waste PET and produce PET functionalized additives. Then, the rheological properties, and chemical properties of the asphalt binders modified with various compositions of PET additives and CR were investigated. Ultimately, it is expected that a value-added recycling method can be developed to help recycle both waste plastic and waste vehicle tyre, while at the same time producing a high-performance bituminous paving mixture.

2. Recycling of Waste PET

Recycling of waste PET can be classified into two major categories, i.e., physical recycling and chemical recycling (Firas et al., 2005). Either process provides a distinct set of advantages that make it particularly beneficial for specific applications or requirements.

Physical recycling is also known as mechanical recycling. It mainly involves the following treatments and operations: 1) separating PET from other plastics, 2) washing to remove dirt and other contaminants, 3) grinding and crushing to reduce the PET particle size, 4) extruding by heat, and 5) pelletisation and reprocessing into new PET products. Compared with chemical recycling, physical recycling is relatively easier to implement and in general requires less investment. However, there are numerous barriers in practice that prevent its common use. For example, paper labels and adhesives that are commonly attached to PET products may cause discoloration; PET containing residual moisture degrades readily when reprocessed; and the collection, sorting and separation costs are high because of the low bulk density of PET bottles and the stringent requirement to have well sorted feedstock. In pavement engineering, some attempts have been made to use physically recycled waste PET in asphalt pavement. In these applications, waste PET was cut into small pieces and directly added to the hot asphalt binder or mixture during production. It was reported that adding scrap waste PET improved the rutting resistance, fatigue resistance, and Marshall stability, and reduced the moisture susceptibility of asphalt binder and mixture (Garcia-Morales et al., 2006). However, PET modified asphalt has not achieved widespread acceptability in practice yet due to the following concerns (Ameri et al., 2016): phase separation of PET modified asphalt mixtures caused the very smooth surface texture of PET, and potential emission and odour during the mixing of untreated PET with hot asphalt binder or mixture. Therefore, an additive form of PET whose surface texture has been degraded through chemical treatment might be more suitable for pavement applications (Padhan et al., 2013, Padhan et al., 2017 Gürü et al., 2014).

Chemical recycling is an alternative of physical recycling which chemically degrades or depolymerizes waste PET before its reuse. Depending on the chemical agent used, different chemical recycling routes can be applied, such as methanolysis, glycolysis, hydrolysis, aminolysis and hydrogenation (Daniel Paszun and Tadeusz Spychaj, 1997). Among various treatment routes, aminolysis of PET waste has been scarcely studied so far and there have been very few applications of this process for PET recycling in commercial scale. Chemical depolymerisation of postconsumer PET through an aminolytic chain cleavage yields corresponding diamides of terephthalic acid (TPA) and ethylene glycol (Shukla and Harad, 2006; Spychaj et al., 2001). Depolymerisation of waste PET using aminolysis can be carried out using different types of amines, such as allylamine, ethanol amine, tri-ethanol amine and polyamines. The products of PET aminolysis reaction have been used in the manufacture of plasticizers, hardeners and rigid polyurethane foams (Parab et al., 2014; Li et al., 2014). In addition, an aminolysis product of PET has also been used for asphalt modification and proven to be a suitable antistripping agent (Padhan et al., 2013). In this research, ethanolamine (EA), which is one of the cheapest and widely available amines, was used for the aminolytic degradation of waste PET. The degradation product after purification was characterized by analytical techniques and used for subsequent modification of asphalt. It was anticipated that this aminolysis product with the terminal amine groups will have a significant effect in improving the performance properties of crumb rubber modified bitumen. The detailed PET aminolysis procedure adopted in this study will be described in the next section.

3. Materials and Experimental Program

3.1 Materials

The CR powders used in this study were produced from local tyres through ambient grinding. The size of the powders used were minus 40 mesh. Local waste plastic bottles were collected as the source for waste PET. Asphalt binder with a penetration grade of 60/70 (Pen 60/70), commonly used in Hong Kong, was used as the base asphalt binder. Ethanolamine (EA) purchased from a local chemical company was used as the chemical agent to degrade waste PET and produce the PET additive.

3.2 Preparation of Waste Plastic Derived Additive

The weight ratio of EA to waste PET was 1:3. The reaction was performed under reflux at a temperature of 130 °C to 140 °C for 8 hours. At the end of the reaction, the PET flakes disappeared, and the mixture became homogeneous. Ice cold distilled water was then used to crystalize the residue, followed by filtration. The filtrate contained mainly unreacted EA and small amount of water-soluble PET degradation products, such as glycol. A white crystalline powder named bis (2-hydroxy ethylene) terephthalamide (BHETA) was finally produced as shown in Figure 1.

Figure 1. BHETA Additive after Aminolysis

3.3 Spectroscopy Analysis and Mechanism Investigation of BHETA Formation

Purified BHETA was characterized by the Fourier Transform Infrared spectroscopy (FTIR) tests and mass spectroscopy analysis. As Figure 2 illustrates, the complete conversion of PET into BHETA was verified by the disappearance of the ester group peak at 1735 cm^{-1} and appearance of new amide peaks at 1637 cm^{-1} and 1547 cm^{-1} in the FTIR spectra. Furthermore, the peaks at 1054 cm^{-1} and 3296 cm^{-1} confirmed the presence of primary alcohols. The mass spectrum in Figure 3 showed a major molecular ion peak at 220, confirming the structure of BHETA derived diamides. The molecular ion peaks at 253 and 252 indicated the presence of the monomer of diamides.

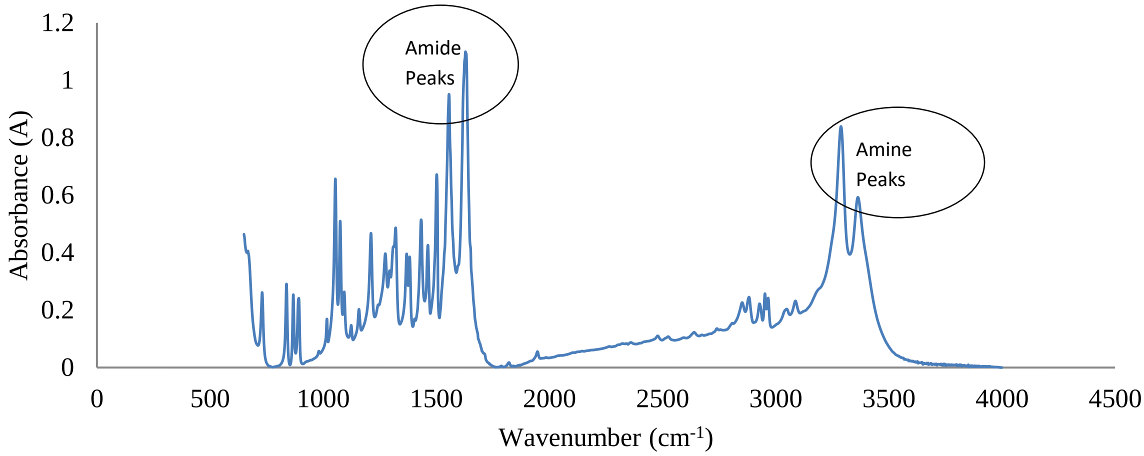


Figure 2. FTIR Spectra of BHETA

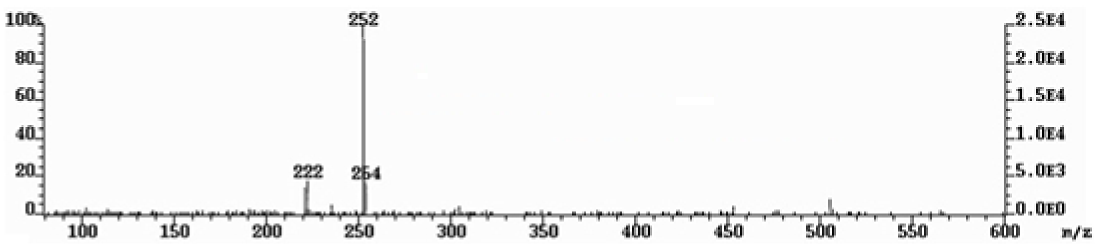
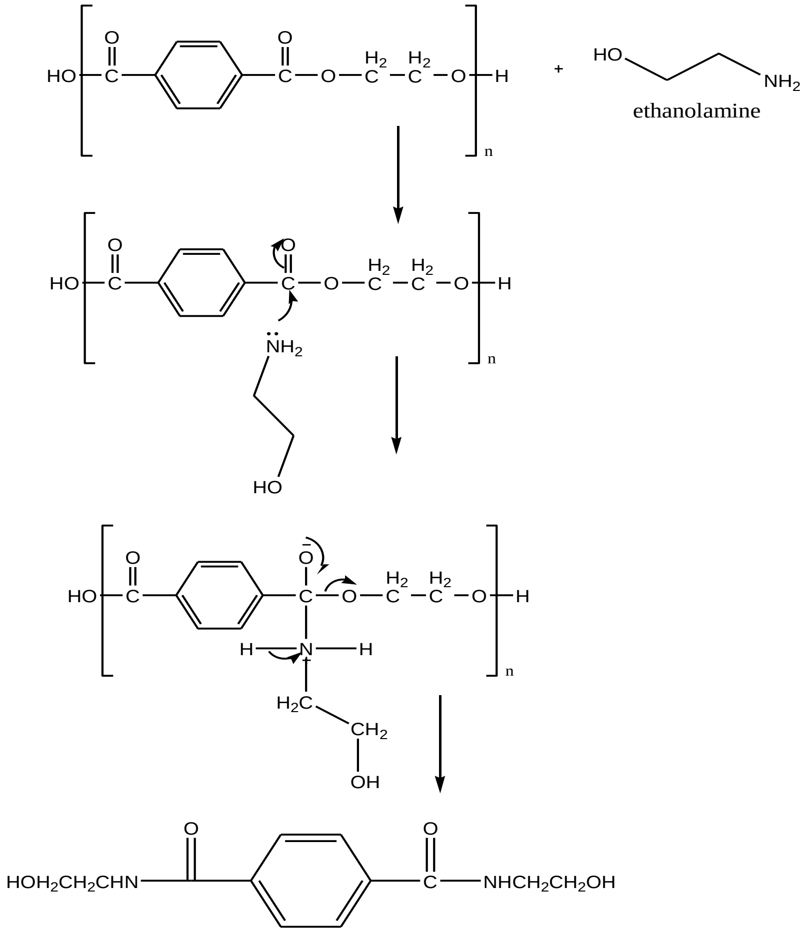


Figure 3. Mass Spectra of BHETA

3.4 Mechanism of Depolymerization of Waste PET

EA has two nucleophilic centres, i.e., nitrogen and oxygen. Due to the electronegative difference between nitrogen and oxygen, there is a lone pair of electrons. Thus, the amine group of EA can attack the ester linkage of PET to form BHETA. The mechanism of the reaction is shown in Figure 4.



BHETA

Figure 4. Mechanism of Aminolysis of PET

3.5 Preparation of Modified Asphalt

To prepare the modified asphalt, Pen 60/70 asphalt binder was first heated at 180 °C in an aluminium container until it became a free-flowing semi-viscous liquid. The sample was then transferred into a blending machine chamber and 10% of CR by weight of virgin binder was added. The asphalt and CR mixture was stirred by applying high-speed shear mixing at 180 °C and at the rate of 5000 rpm for 1 hour to produce CRMA (10CR). Finally, 3, 5, and 7 wt.% of BHETA was mixed with 10% of CR at 5000 rpm for 1 hour at 180 °C to produce 10CR-3BH, 10CR-5BH and 10CR-7BH, respectively.

3.6 Rheological and Chemical Characterization of Asphalt Binder

All asphalt blends were first subjected to the basic rheological property tests, including penetration (ASTM D5), softening point (ASTM D36) and viscosity (ASTM D4402). High temperature storage stability tests were then performed in accordance with ASTM D 5644. Superpave rheological measurements were finally conducted with a Dynamic Shear Rheometer (DSR) (Physica MCR302, Anton Paar). Within the linear viscoelasticity region, the frequency sweep tests at 60 ^{o}C were performed over a frequency range from 0.1 to 10 Hz using 25-mm-diameter DSR plates with 1 mm gap. The multiple stress creep recovery (MSCR) tests were conducted at two different temperatures (70 ºC and 76 ºC) in accordance with AASHTO T350-14. Material characterization tests were first conducted using Fourier transform infrared spectroscopy (FTIR) to identify the functional groups in the modified binders. Subsequently, thermal characterization using a thermogravimetric analyzer (TGA) was performed to study the thermal stability of the various binders. Figure 5 shows the experimental program of this study. All samples were tested in triplicate.

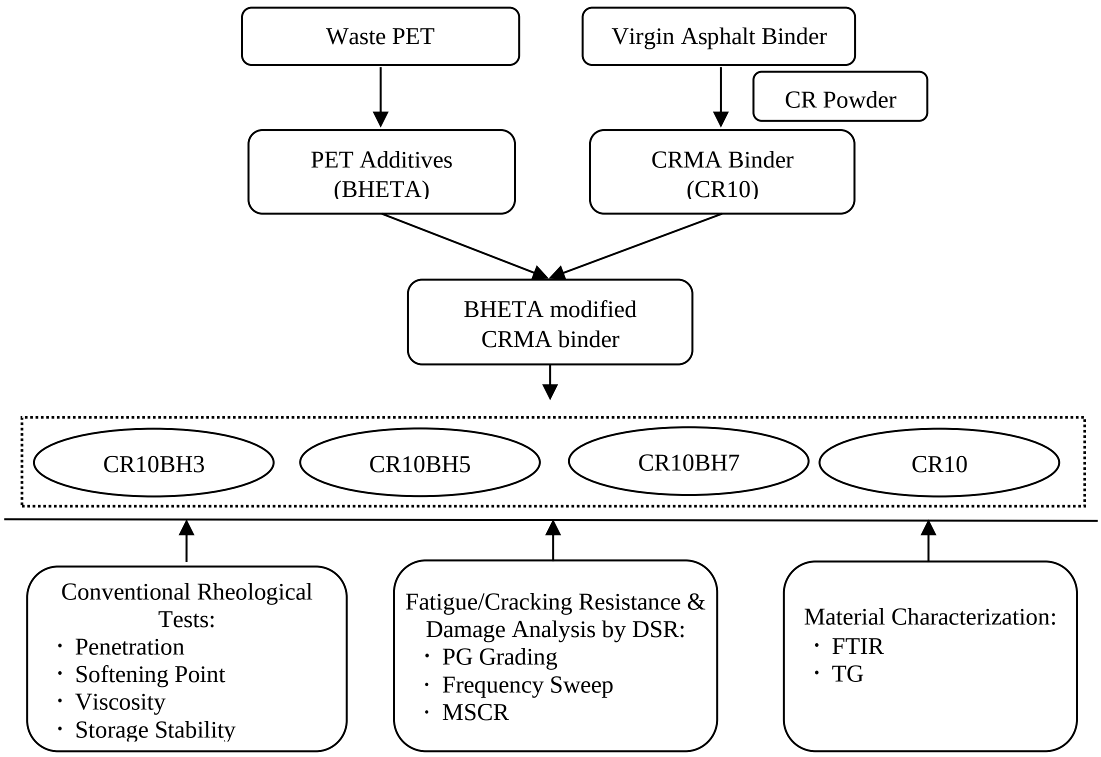


Figure 5. Experimental Program

4. Results and Discussion

4.1 Conventional Rheological Properties

4.1.1 Penetration and softening point

The results of the penetration and softening point tests are shown in Figure 6. The penetration tests were conducted at 25°C. It can be observed that compared to the base CRMA binder (without BHETA additive), the CRMA binders with BHETA have lower penetration values and the penetration values decrease with the increase of the BHETA percentage. The penetration values were seen to decrease on average of around 5% with the addition of PET additive at 3%, 5% and 7% dosages respectively. Similarly, compared to the base CRMA binder, the CRMA binders with BHETA have higher softening points and the softening points increase on average of around 6% with the increase of the BHETA percentage.

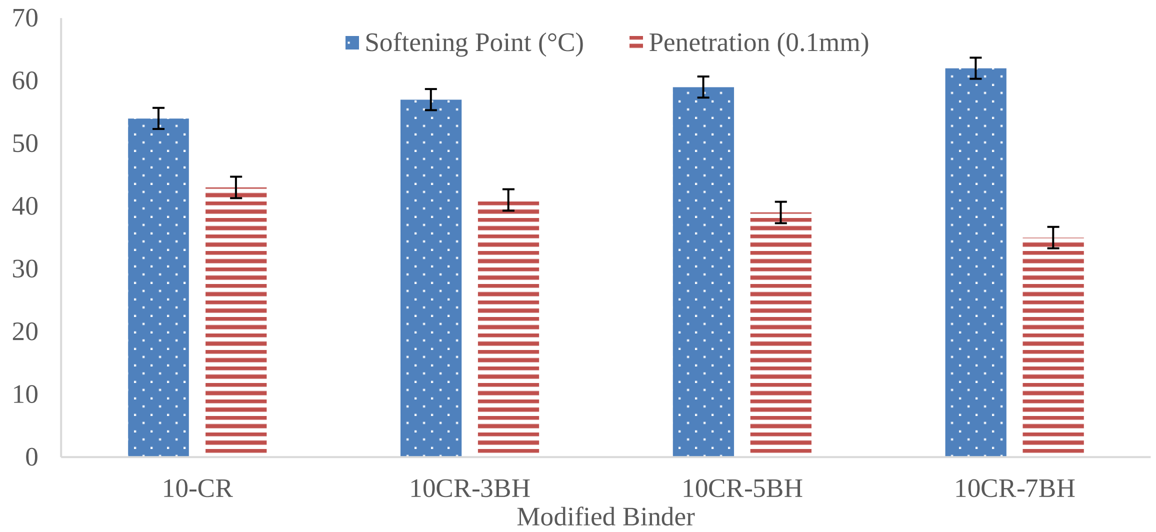


Figure 6. Softening Point and Penetration Results

4.1.2 Viscosity

The Brookfield viscosity test was carried out to assess the workability of the CRMA binders. The viscosity tests were conducted at three temperatures: 150 ºC, 165 ºC and 180 ºC. From the results presented in Figure 7, it can be seen that the viscosities of all CRMA binders decrease with the increasing test temperature. In general, the addition of the BHETA additive increases the viscosity of CRMA binder at high temperatures. When the BHETA additive is added at small percentages (3% to 5%), the increase in viscosity is relatively small, i.e. within 20% of the base CR binder. However, when 7% of BHETA additive is added, there is a significant increase in viscosity of about 66% at the mixing temperature of 180 ^{o}C. This indicates that there is a maximum additive percentage which complies with both the desired workability and rheological performance. Warm mix asphalt (WMA) technologies can be considered and applied to address the workability concern as needed.

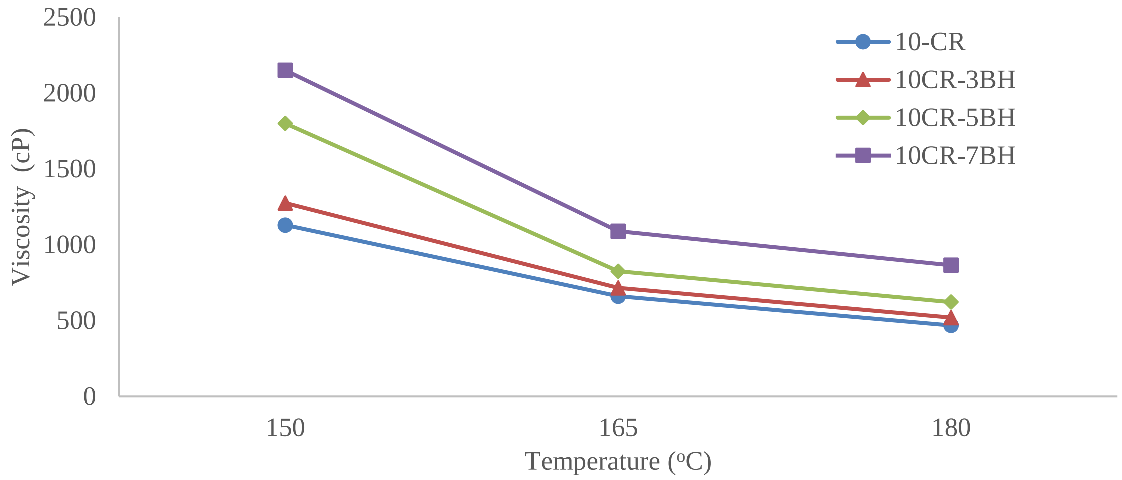


Figure 7. Effect of BHETA on Viscosity

4.1.3 Storage stability

To characterize the storage stability of the modified binders, the heated binder sample was first poured into an aluminium toothpaste tube (32 mm in diameter and 160 mm in height). Then the tube was sealed and stored vertically in an oven at 163 ^{o}C for 48 hours, removed from the oven, cooled to room temperature and cut into three equal parts. The samples taken from the top and bottom parts were used to evaluate the storage stability of CRMA based on their measured softening points. If the difference in the softening point between the top and bottom sections is less than 4 ^{o}C, the sample is considered to have acceptable high-temperature storage stability for crumb rubber modified binders (Padhan et al., 2017; Biro et al.,2005). If the softening points differ by more than 4 ^{o}C, the sample is considered unstable. As shown in Figure 8, the base CRMA exhibited significant phase separation at high temperatures, evidenced by the high softening point difference (SPD) of 10 ^{o}C. The addition of the BHETA additives effectively reduced the SPD in the CRMA. When 7% BHETA additive was added, the SPD was decreased to 4 ^{o}C. This could be attributed to the chemical and physical interaction between CR and BHETA, as well as the denser polymer network within the asphalt matrix which prevents the modified CR from phase separation.

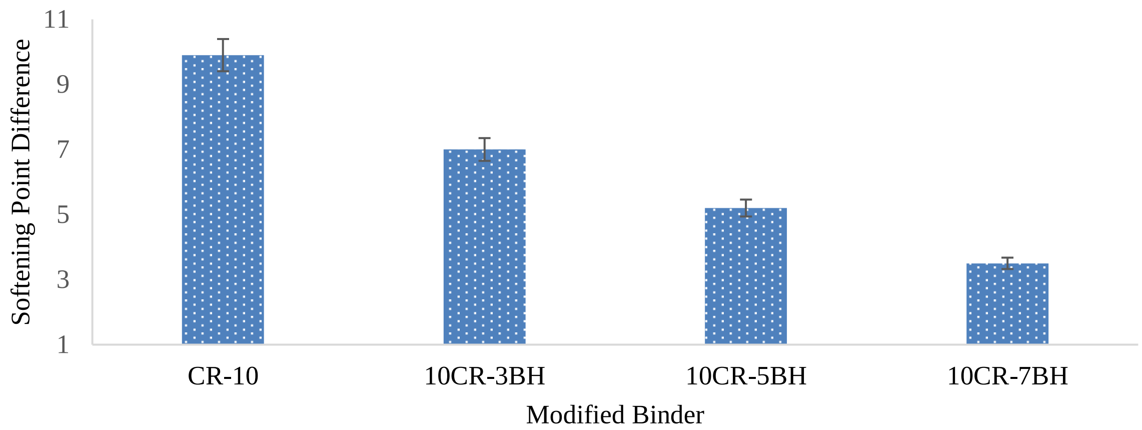


Figure 8. Effect of BHETA on Storage Stability of CRMA Binder

4.2 Rutting and Fatigue Properties

DSR tests were carried out to further assess the rutting and fatigue properties of CR and BHETA modified asphalt at high and intermediate temperatures, respectively. The testing temperatures were selected as 64 ^{o}C, 70 ^{o}C, 76 ^{o}C and 82 ^{o}C to examine the rutting parameter ($G^*/Sin(\delta)$) in accordance with ASTM D2872. The intermediate testing temperature range was selected as 16 ^{o}C to 25 ^{o}C at 3 ^{o}C increments to examine the fatigue factor ($G^*Sin(\delta)$) in accordance with ASTM D6521. The rutting factor test results for unaged and rolling thin-film oven (RTFO) aged binders are shown in Figure 9 and Figure 10, respectively. According to the Superpave specification, AASHTO: MP1, the $G^*/Sin(\delta)$ value should be at least 2.2 kPa for RTFO aged asphalt and 1.0 kPa for neat asphalt at the maximum pavement design temperature.

The results in Figures 9 and 10 indicated that for both unaged and RTFO aged binders, G*/sin(δ) values of 10CR-3BH, 10CR-5BH and 10CR-7BH blends are marginally higher. In other words, adding BHETA led to better rutting resistance of the CRMA. G*sin(δ) is the fatigue resistance parameter defined by Superpave asphalt binder specification. It should have a maximum value of 5000 kPa for pressurised ageing vessel (PAV) aged asphalt samples. From Figure 11, it can be seen that all modified binders met the fatigue criterion at 19 ^{o}C. In addition, BHETA modification was found to generally decrease the G*sin(δ) value of the CRMA. At an additive dosage of 5%, the G*sin(δ) values decreased by around 8% at the failure temperature of 16 ^{o}C. Therefore, it can be concluded that BHETA modified CRMA offered better fatigue resistance than the base CRMA.

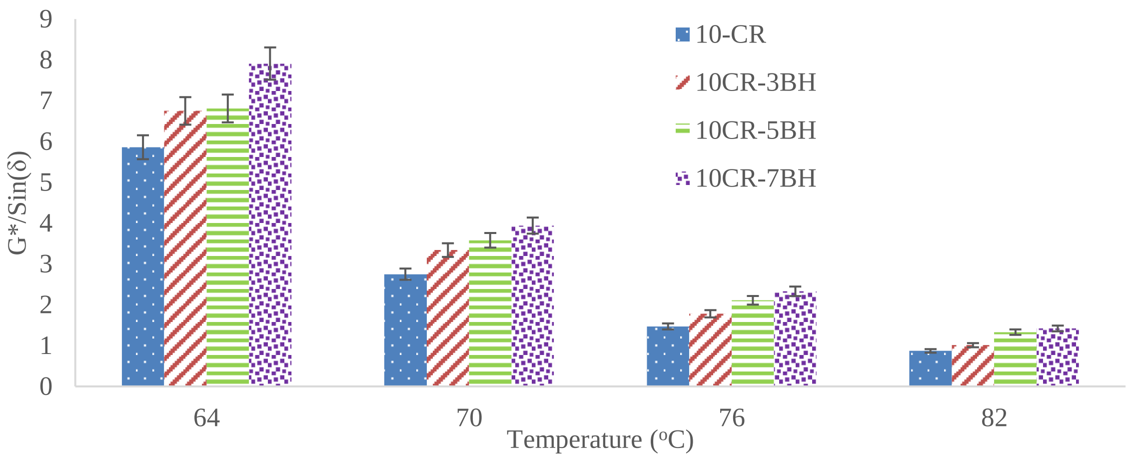


Figure 9. Effect of BHETA on Rutting Factor (G*/Sin(δ)) of Unaged CRMA Samples

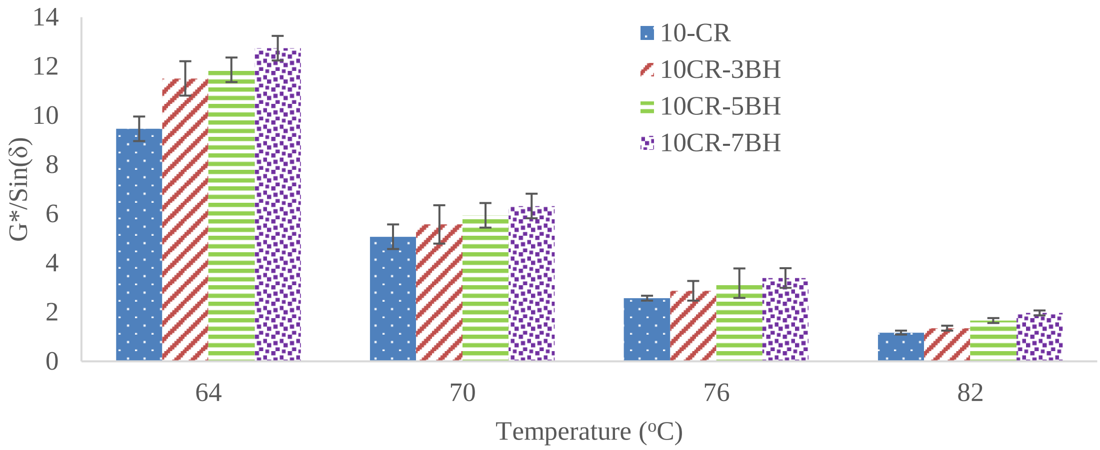


Figure 10. Effect of BHETA on Rutting Factor (G*/Sin(δ)) of RTFO Aged CRMA Samples

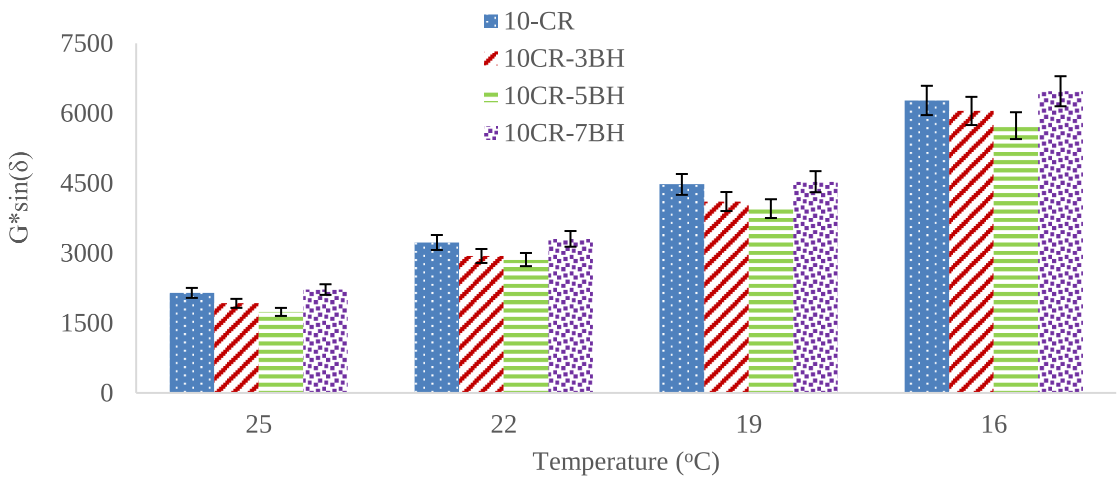


Figure 11. Effect of BHETA on Fatigue Factor (G*Sin(δ)) of PAV Aged CRMA Samples

4.3 Frequency Sweep Test

The frequency sweep tests were carried out using the DSR at a range of frequencies (0.01 to 600 rad/sec). All the tests were conducted with the 25 mm DSR plate diameter and 1 mm plate gap at 60 ^{o}C. Earlier studies have indicated that the frequency sweep tests at several frequencies could be used to categorize the linear viscoelastic response of the binders (Airey et al., 2008). As Figure 12 shows, the three binders with BHETA additives generally have similar viscoelastic properties. The increasing frequency results in an increase of complex modulus and a reduction of phase angle. As the test frequency increases from 10 to 100 Hz, the increase of |G*| value is more noticeable. In Figure 13, the phase angle has an increase initially (noticeable for some binders) and then reduces very quickly. In general, the CRMA binders modified with BHETA additives have higher |G*| values and lower δ values (above 50 rad/s) than the base CRMA. It is evident from these results that BHETA modified CRMA would provide better rutting resistance as compared to the base CR binder.

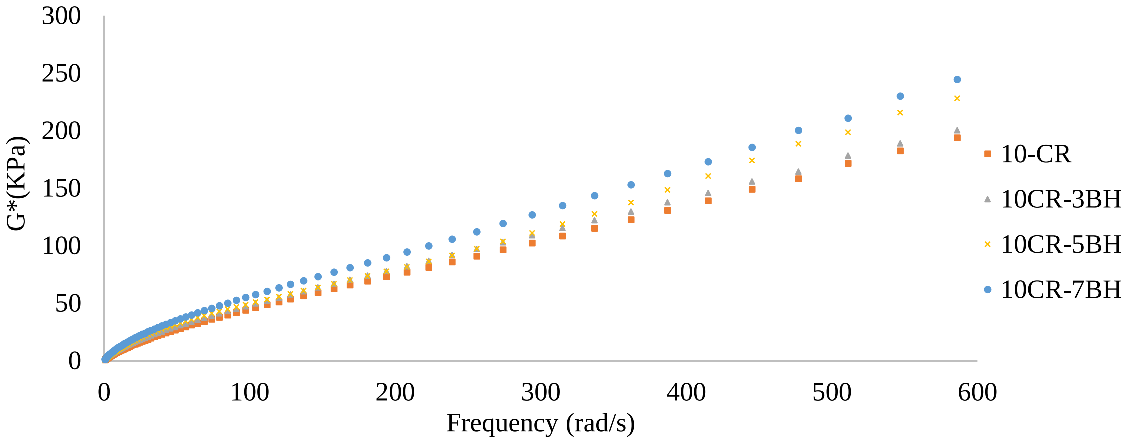


Figure 12. Shear Modulus (G*) vs Frequency at 60°C

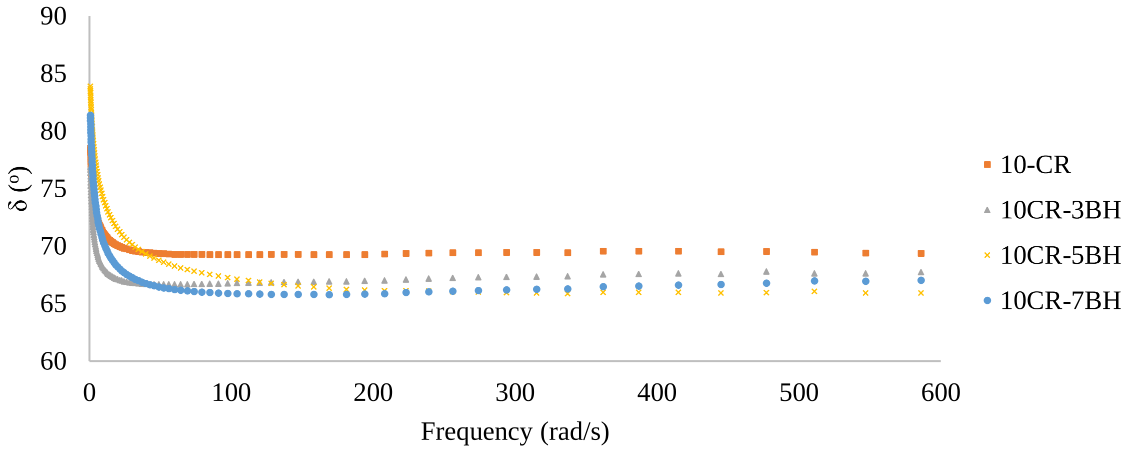


Figure 13. Phase Angle (δ) vs Frequency at 60°C

4.4 MSCR test

MSCR test can be used to characterize the recoverable strain (elastic responses) and J_{nr} (non-recoverable creep compliance) of polymer modified asphalt binders, which is reportedly more accurate than the conventional DSR tests (Angelo et al., 2009). The tests were conducted on both the conventional CRMA (10-CR) and the CR-BHETA modified asphalt to identify the non-recoverable creep compliance (J_{nr}) (Figure 14) and elastic response (Figure 15) at two different stress levels, 0.1 kPa^{-1} and 3.2 kPa^{-1}. As shown in Figure 15, the percentages of elastic recovery of CR-BHETA modified binders were significantly greater than that of the 10-CR binder at 70°C and 76 °C. Furthermore, the J_{nr} values for all modified CRMA binders were lower than that of the 10-CR binder (Figure 14). J_{nr} values of BHETA modified CRMA binders increase with the increase of the BHETA content. The increase in elastic response of CRMA with BHETA additives may not be necessarily be due to just physical interaction because BHETA has been previously used for plasticizer applications (Parab et al., 2014). Therefore, it is likely that there is some chemical interaction between BHETA, CR and asphalt. Overall, it is apparent that the addition of the BHETA additive would provide more elasticity and flexibility to the asphalt binder to recover from deformation under heavy traffic load.

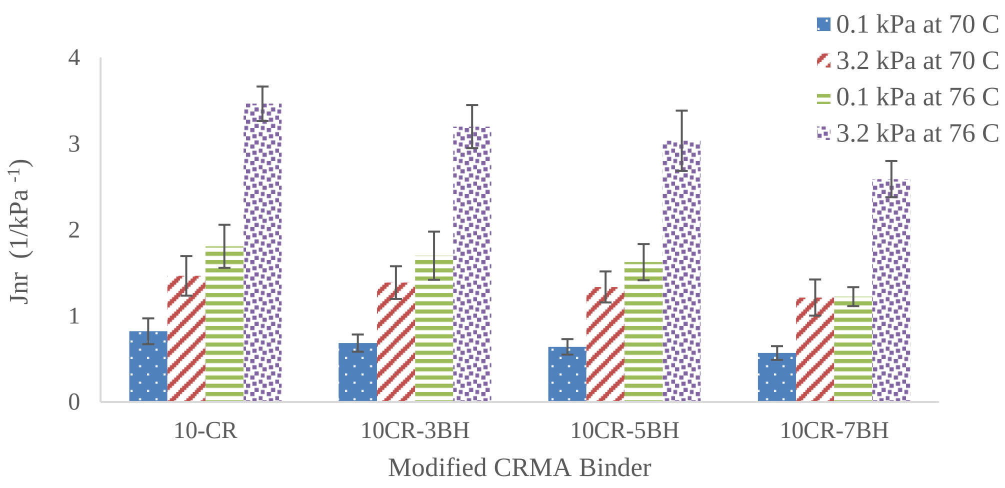


Figure 14. Effect of BHETA on Non-Recoverable Creep Compliance (J_{nr})

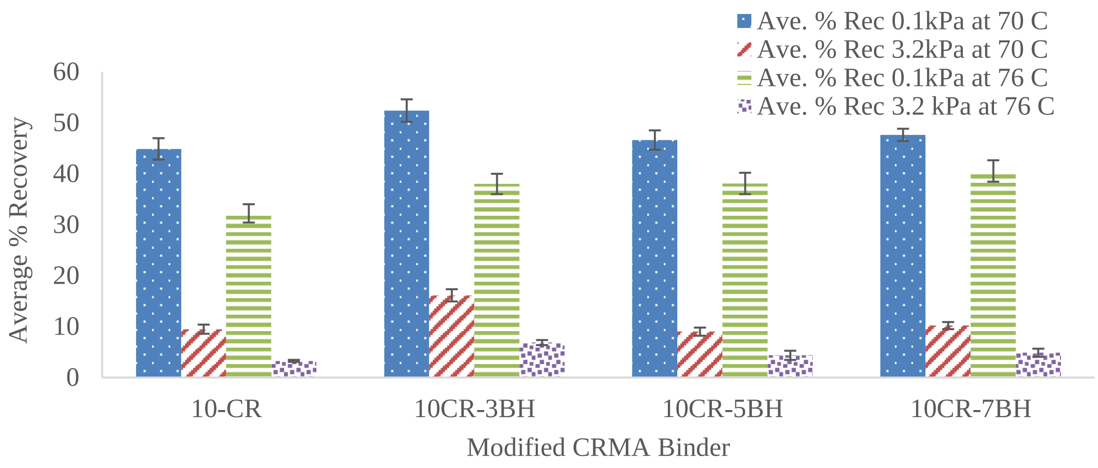


Figure 15. Effect of BHETA on Average % Recovery

4.5 FTIR Test

The effects of crumb rubber and BHETA on the chemical compositions of asphalt were evaluated using FTIR tests. Figure 16 shows the FTIR spectra of all asphalt binders. The bands observed at around 2919 cm^{-1} and 2850 cm^{-1} were due to Alkyl C–H_{str} and Aliphatic C–H_{str} stretches, respectively, while those at 1600 cm^{-1} and 1012 cm^{-1} can be attributed to the C-C_{str} and C–O_{str} stretches (Hossain et al., 2012; Fang et al., 2008). CRMA binders containing BHETA additives show different peaks at 1645 cm^{-1}, 1550 cm $^{-1}$ and 3300 cm $^{-1}$ to 3400 cm $^{-1}$ compared with the base CRMA binder. This clear shifting of peaks may be due to the chemical interaction between BHETA and crumb rubber. It is hence likely that the terminal amine groups interact within the asphalt-crumb rubber matrix to form more complex microstructures with chemical linkages. Further studies should be conducted to ascertain the exact interaction mechanism.

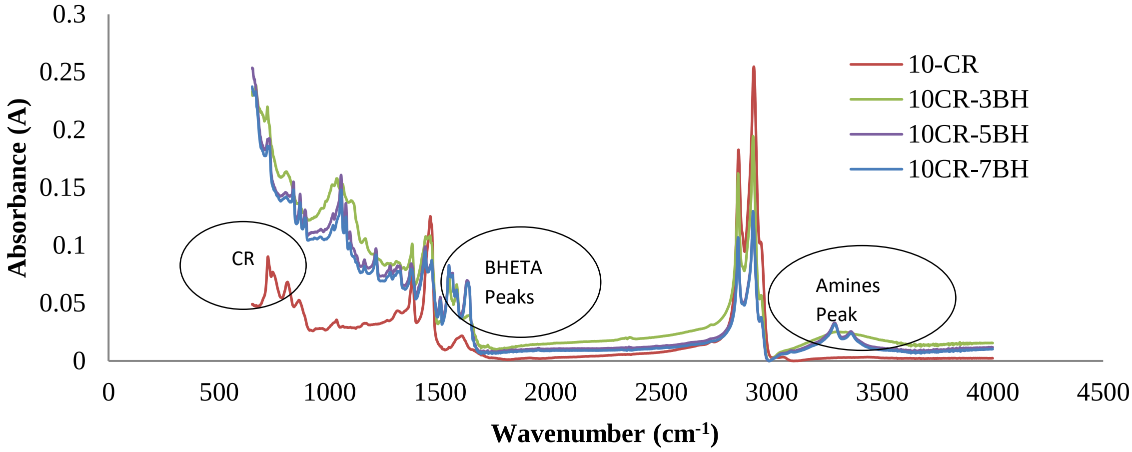


Figure 16. FTIR Spectra of Binder Samples

4.6 Thermogravimetric Analysis (TGA)

The thermal stability of CRMA binder is an important property to be considered in the analysis of the structural changes of modified asphalt (Chen and Qian, 2003; Gawel et al., 2006). In this research, the thermal behaviours of 10CR-3BH, 10CR-5BH and 10CR-7BH binders were studied by TGA at the heating rate of 20 ^{o}C/min. The TG curves for each modified asphalt binder are shown in Figure 17. It can be seen that all binders undergo major loss of mass percentage between 350 ^{o}C and 550 ^{o}C, which is mainly due to the loss of the light asphalt components, such as saturates and aromatics, and the decomposition of CR particles and BHETA. It can also be noticed that the base CRMA and the BHETA modified CRMA have similar temperature range for main decomposition (approximately 300-500 ^{o}C), indicating limited effect of incorporating BHETA on the thermal stability of CRMA.

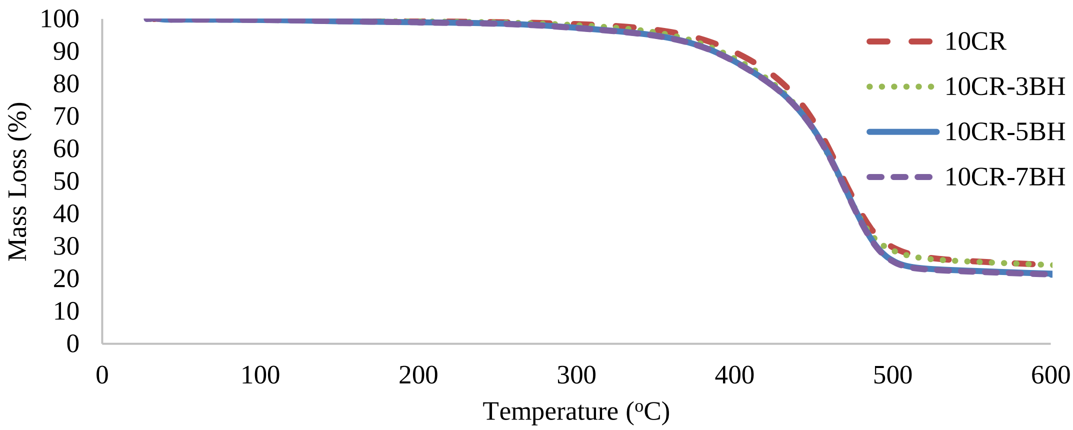


Figure 17. TG Analysis Curve of 10-CR, 10CR-3BH, 10CR-5BH and 10CR-7BH

5. Findings and Recommendations

In this study, a series of rheological property and chemical analysis tests were carried out to characterize the physical and chemical properties of CR-BHETA modified asphalt binders. Based on the test results, the following findings have been obtained:

- The results of the penetration and softening point tests showed that the addition of BHETA additives increased the stiffness of CRMA binder.
- The CRMA binders containing the BHETA additives showed better storage stability, which would enable it to be used in paving applications after prolonged storage at high temperatures.
- The viscosity of CRMA binder increased after the addition of BHETA additives. Hence, there is a maximum percentage of additive to ensure the workability of the modified binder. Warm mix asphalt additives may be considered and used to improve the workability of the modified binder.
- The PG grading and frequency sweep test results demonstrated that BHETA modified CRMA had better rutting resistance than the base CRMA binder.

- The MSCR test results showed that the BHETA modified CRMA binders had better elastic and recovery properties as compared to the base CRMA binder.
- The BHETA modified CRMA binders also offered improved fatigue resistance than the base CRMA binder.
- The FTIR analysis results showed that the effects of BHETA on CRMA binder properties may be due to both physical and chemical interaction.
- The thermal analysis results indicated that there is limited or negligible effect of incorporating BHETA into CRMA in terms of thermal stability.

Overall, the incorporation of BHETA provided positive effects on the rheological properties of CRMA binders. The usage of such PET based additives as a modifier for CRMA represents an innovative approach to deal with a relevant waste recycling problem while simultaneously recovering two value-added materials. A limitation of this study was that only binder tests were carried out; therefore, it is suggested that further studies be conducted to investigate the performance of the bituminous mixtures prepared with the CR-BHETA modified binder. In addition, life-cycle assessment and cost analysis studies need to be undertaken to determine practical and economic implications of using such additives in paving applications.

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